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Review

Flavonoid Composition of *Citrus* Juices

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Abstract: In the early nineties the presence of flavonoids in *Citrus* juices began to attract the attention of a number of researchers, as a result of their biological and physiological importance. This short review will explore two different aspects. The first part will focus on analytical techniques for the characterization of juices from different *Citrus* fruits regarding their flavonoid content (even if present in only trace amounts), concentrating on the most widely used methods (LC-MS and LC-MS-MS). The second part analyzes data reported in the literature regarding the composition of *Citrus* juices. The main components that have been detected so far are flavanone-*O*-glycosides and flavone-*O*- or -*C*-glycosides. The presence of such derivatives in various hand-squeezed and industrial juices is discussed, with special emphasis on their correlation to different species.

Keywords: *Citrus*, flavonoids, *Citrus* juices, HPLC, HPLC-MS

Introduction

Plant flavonoids [1] are a large group of very different compounds sharing the common feature of phenol moieties. They are, with a few notable exceptions, plant metabolites deriving from the shikimate pathway and the phenylpropanoid metabolism [2]. Flavonoids are aromatic secondary plant metabolites, which have been recognized as important due to their physiological [3] and pharmacological [4] role and their health benefits [5]. The amount of interest they have attracted is demonstrated by more than 13,000 articles and books dealing with various aspects of flavonoid chemistry, biochemistry, pharmaceutical and nutritional role which have appeared since 1990.

Flavonoids show a strong antioxidant and radical scavenging activity [6] and appear to be associated with reduced risk for certain chronic diseases [7], the prevention of some cardiovascular disorders [8] and certain kinds of cancerous processes [9]. Flavonoids exhibit also antiviral [10], antimicrobial [11], and anti-inflammatory activities [12], beneficial effects on capillary fragility [13] and an ability to inhibit human platelet aggregation [14], antiulcer [15] and antiallergenic [16] properties. However, the actual *in vivo* mechanism of action is largely unknown. One of the reasons is that most studies have focused on *in vitro* tests at doses or concentrations much higher than those documented in humans, whereas few clinical investigations have been carried out on biomarkers of some of the diseases mentioned above, but results have been contradictory [17]. Epidemiological studies have shown an inverse association between risk and intake level of some particular flavonoids, but further clinical trials are needed to assess a more precise correlation between the level of flavonoids consumption and human health benefits [18]. However, the probable mechanism by which they act, and the potential clinical applications have been reviewed [19].

Flavonoids are frequently found in fruits [20], vegetables and cereals [21]. Over the past few years investigations into flavonoids from dietary sources have attracted an ever growing interest as a consequence of their well-established versatile health benefits [22]. Food preparation and processing of fresh fruits and vegetables may decrease flavonoids content by 50% owing to their leaching into water, or by removal of the richest parts of the plant [23]. In order to overcome this problem, and even more to make processed foodstuffs a better vehicle for flavonoid intake, a considerable amount of work has been done on the modification of their biosynthesis in plants. Recent advances in bioengineering have been extensively reviewed [24].

For the time being, the main source of flavonoid intake remains dietary intake. This accounts for an average daily intake of flavonoids between 70 and 170 mg/day, the difference mostly depending on dietary and cultural habits. *Citrus* fruits and *Citrus* juices stand out among the most common phenolic-rich dietary sources [25]. The most significant cropped *Citrus* fruits are oranges, mandarins, grapefruits and acid *Citrus* fruits, namely lemons, bergamots and limes.

Fresh fruits and their hand-squeezed or industrially processed juices, contain mostly flavanones and flavones. Although more than 5000 flavonoid derivatives have been characterized [26], only a limited number of representative derivatives have been found and identified. Many others are often only present in very low concentrations and have yet to be identified. Their significance, however, may outweigh their simple concentration levels.

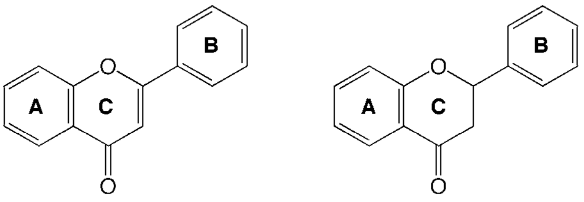
In general, flavonoids may contribute to fruit and juice quality in many ways, influencing the appearance, the taste and the nutritional value of the product from the plant. In lemon and orange juices, for instance, hesperidin can contribute to the formation of sediments which result in undesirable cloudiness [27], whereas naringin markedly influences the bitterness of the juice of grapefruits and bergamots [28].

This short review examines the data in the literature regarding the flavonoid content of *Citrus* juices, particularly over the last fifteen years, and focuses on the juices of the most widespread *Citrus* species.

Citrus juice flavonoids

A flavonoid skeleton is composed of two aromatic rings (commonly designated as A and B), which are connected through a pyrone ring (C) in the case of flavones, or a dihydropyrone ring in the case of flavanones, as shown in Figure 1.

Figure 1. Flavone and flavanone skeletons.



Flavone skeleton Flavanone skeleton

Flavonoids are mainly present in *Citrus* fruits as their glycosyl derivatives. Aglycones (the forms lacking the sugar moieties) occur less frequently in juices, owing to their lipophilic nature and hence their low solubility in water. The presence of a relatively large number of flavonoids in *Citrus* juices is a result of the many different combinations that are possible between polyhydroxylated aglycones and a limited number of mono- and disaccharides. The most common sugar moieties include D-glucose and L-rhamnose. The glycosides are usually *O*-glycosides, with the sugar moiety bound generally to the aglycone hydroxyl group at C-7, or at the C-3 in some cases. In addition to these, *C*-glycosides have also been detected in various *Citrus* fruits or juices.

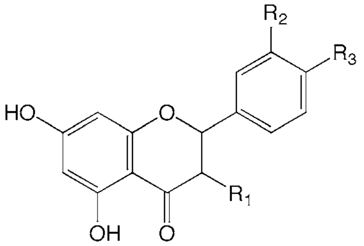


Table 1. Flavanone aglycones.

	Compound name	R_1	R_2	R_3
1	Hesperetin	H	OH	OMe
2	Naringenin	H	H	OH
3	Taxifolin	OH	OH	OH
4	Isosakuranetin	H	H	OMe
5	Eriodictyol	H	OH	OH

All the flavonoid species that have been so far identified and quantified in *Citrus* juice are reported in Tables 1-5. Table 1 presents the flavanone aglycones of recovered glycosides in *Citrus* juices. All these aglycones have a skeleton in which two hydroxyls are present at the C-5 and C-7 positions. In hesperetin (**1**) and isosakuranetin (**4**) the C-4’ position is methoxylated. The flavanone taxifolin (**3**) contains a hydroxyl group in the C ring C-3 position, and can thus also be classified as a flavanol. It is worth mentioning that it is the only flavanol that has been detected in *Citrus* juices to date.

Flavone aglycones are summarized in Table 2. Acacetin (**6**) and diosmetin (**12**) present a methoxy moiety at C-4’ position, whereas in chrysoeriol the methoxyl group is bound to C-3’. Kaempferol (**9**) and quercetin (**10**) both bear hydroxyl group at the 3 position and are often referred to as flavonols.

Table 3 shows the group of compounds classified as polymethoxyflavones (PMFs). These are usually found as components of the essential oils fraction of *Citrus* peels [29]. Hand-squeezed juices contain no detectable traces of this class of compounds [30]. Commercial juices, on the other hand, are rich in PMFs because the industrial processing of fruits leads to juices being contaminated with the peel constituents (*vide infra*).

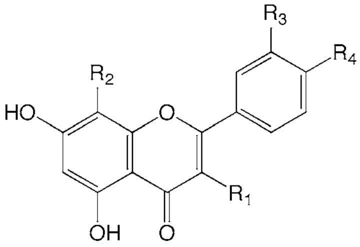


Table 2. Flavone aglycones.

	Compound name	R_1	R_2	R_3	R_4
6	Acacetin	H	H	H	OMe
7	Isoscutellarein	H	OH	H	OH
8	Luteolin	H	H	OH	OH
9	Kaempferol	OH	H	H	OH
10	Quercetin	OH	H	OH	OH
11	Apigenin	H	H	H	OH
12	Diosmetin	H	H	OH	OMe
13	Chrysoeriol	H	H	OMe	OH

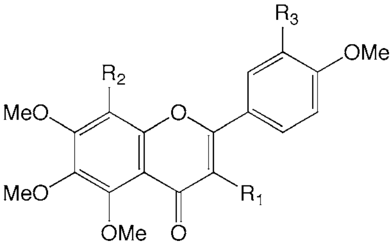


Table 3. Polymethoxyflavones.

	Compound name	R_1	R_2	R_3
14	Quercetogetin	OMe	H	OMe
15	3,3’,4’,5,6,7,8-Heptamethoxyflavone	OMe	OMe	OMe
16	Natsudaidain	OH	OMe	OMe
17	Nobiletin	H	OMe	OMe
18	Sinensetin	H	H	OMe
19	Tangeretin	H	OMe	H
20	Tetramethylscutellarein	H	H	H

The flavanone *O*-glycosides found so far in juices are listed in Table 4. These derivatives have a glycosyl substitution exclusively at the C-7 position (on ring A). Furthermore, only two disaccharides have so far been identified in this group of compounds, and both are L-rhamnosyl-D-glucosyl derivatives: rutinose, which presents a α-1,6 interglycosidic linkage, and neohesperidose, in which the two sugars are linked via a α-1,2 interglycosidic bond (Figure 2).

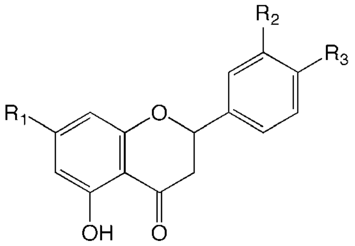
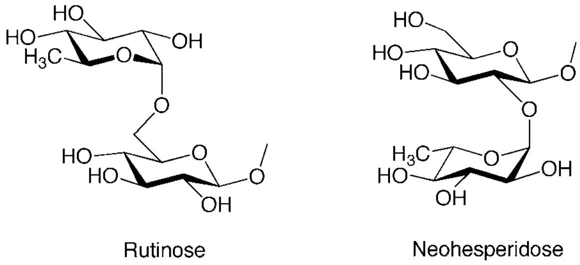


Table 4. Flavanone-*O*-glycosides.

	Compound name	R$_1$	R$_2$	R$_3$
21	Isosakuranetin 7-*O*-rutinoside (Didymin, Neoponcirin)	*O*-Ru[a]	H	OMe
22	Eriodictyol 7-*O*-rutinoside (Eriocitrin)	*O*-Ru[a]	OH	OH
23	Hesperetin 7-*O*-rutinoside (Hesperidin)	*O*-Ru[a]	OH	OMe
24	Naringenin 7-*O*-neohesperidoside (Naringin)	*O*-Nh[b]	H	OH
25	Naringenin 7-*O*-rutinoside (Narirutin)	*O*-Ru[a]	H	OH
26	Hesperetin 7-*O*-neohesperidoside (Neohesperidin)	*O*-Nh[b]	OH	OMe
27	Eriodictyol 7-*O*-neohesperidoside (Neoeriocitrin)	*O*-Nh[b]	OH	OH
28	Isosakuranetin 7-*O*-neohesperidoside (Poncirin)	*O*-Nh[b]	H	OMe

[a] *O*-Rutinose; [b] *O*-Neohesperidose.

Figure 2. Rutinose and neohesperidose.



Rutinose Neohesperidose

Flavone *O*-glycosides found in *Citrus* juices are generally 7-*O*-rutinosides or 7-*O*-neohesperidosides (Table 5), although a 3-*O*-rutinoside has also been reported, namely rutin (**39**). *Citrus* juices also contain a large number of di-*C*-glycosides, along with smaller amounts of mono-*C*-glycosides. For these compounds, substitution is generally on either the C-6 or the C-8, or on both positions.

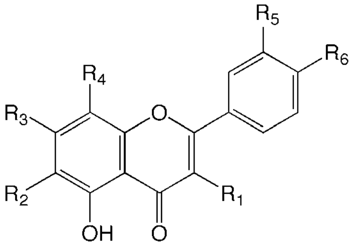


Table 5. Flavone-*C*-glucosides and flavone-*O*-glycosides.

	Compound name	R_1	R_2	R_3	R_4	R_5	R_6
29	Luteolin 6,8-di-*C*-glucoside (Lucenin-2)	H	Glu	OH	Glu	OH	OH
30	Apigenin 6,8-di-*C*-glucoside (Vicenin-2)	H	Glu	OH	Glu	H	OH
31	Chrysoeriol 6,8-di-*C*-glucoside (Stellarin-2)	H	Glu	OH	Glu	OMe	OH
32	Diosmetin 6,8-di-*C*-glucoside (Lucenin-2 4´-methyl ether)	H	Glu	OH	Glu	OH	OMe
33	Apigenin 7-*O*-neohesperidoside-4´-glucoside (Rhoifolin 4´-glucoside)	H	H	*O*-Nh[b]	H	OH	*O*-Glu
34	Chrysoeriol 7-*O*-neohesperidoside-4´-glucoside	H	H	*O*-Nh[b]	H	OMe	OH
35	Apigenin 6-*C*-glucoside (Isovitexin)	H	Glu	OH	H	H	OH
36	Luteolin 7-*O*-rutinoside	H	H	*O*-Ru[a]	H	OH	OH
37	Chrysoeriol 8-*C*-glucoside (Scoparin)	H	H	OH	Glu	OMe	OH
38	Diosmetin 8-*C*-glucoside (Orientin 4´-methyl ether)	H	H	OH	Glu	OH	OMe
39	Quercetin 3-*O*-rutinoside (Rutin)	*O*-Ru[a]	H	OH	H	OH	OH
40	Apigenin 7-*O*-neohesperidoside (Rhoifolin)	H	H	*O*-Nh[b]	H	OH	OH
41	Apigenin 7-*O*-rutinoside (Isorhoifolin)	H	H	*O*-Ru[a]	H	OH	OH
42	Chrysoeriol 7-*O*-neohesperidoside	H	H	*O*-Nh[b]	H	OMe	OH
43	Diosmetin 7-*O*-rutinoside (Diosmin)	H	H	*O*-Ru[a]	H	OH	OMe
44	Diosmetin 7-*O*-neohesperidoside (Neodiosmin)	H	H	*O*-Nh[b]	H	OH	OMe

[a] *O*-Rutinose; [b] *O*-Neohesperidose.

To date, only D-glucosyl derivatives have been characterized. An example of a C-6-glucosylated flavone is isovitexin (**35**), whereas scoparin (**37**) and orientin 4’-methyl ether (**35**) are C-8-substitued flavones. Two glucose moieties are present at the C-6 and C-8 positions of lucenin-2 (**29**), vicenin-2 (**30**), stellarin-2 (**31**), and lucenin-2 4’-methyl ether (**32**).

Analytical Methods

UV, MS, and NMR spectra of Citrus flavonoids

The UV spectra of flavones and related glycosides show two strong absorption peaks commonly referred to as band I (300-380 nm) and band II (240-280 nm). Band I is associated with the presence of a B-ring cinnamoyl system. Band II absorption is due to an A-ring benzoyl system. Substitutions on the A or B ring may produce hypsochromic or bathocromic shifts of the absorptions, which are useful for clarifying structures [31].

Figure 3. UV Spectra of quercetin (**10**, red trace), 3-*O*-glucosyl quercetin (green trace), and 3,4'-di-*O*-glucosyl quercetin (black trace).

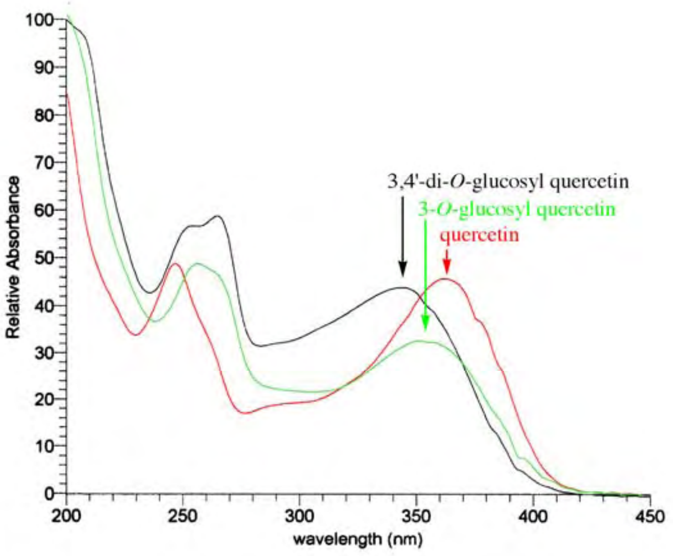


Figure 4. UV Spectrum of the flavanone hesperidin (**23**).

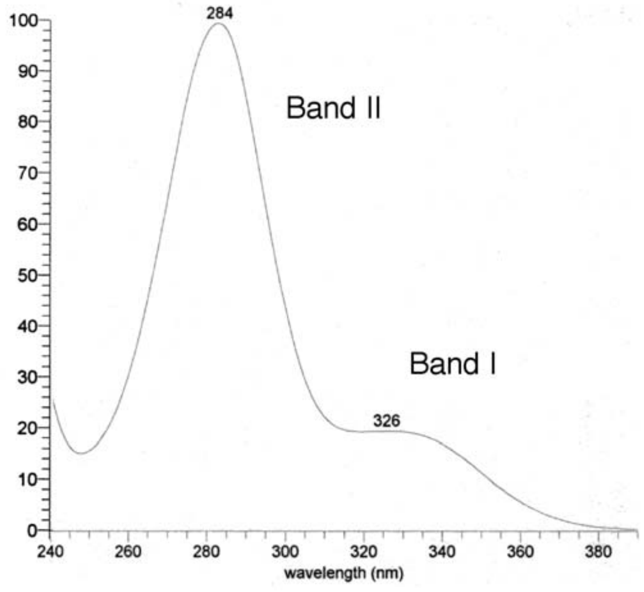
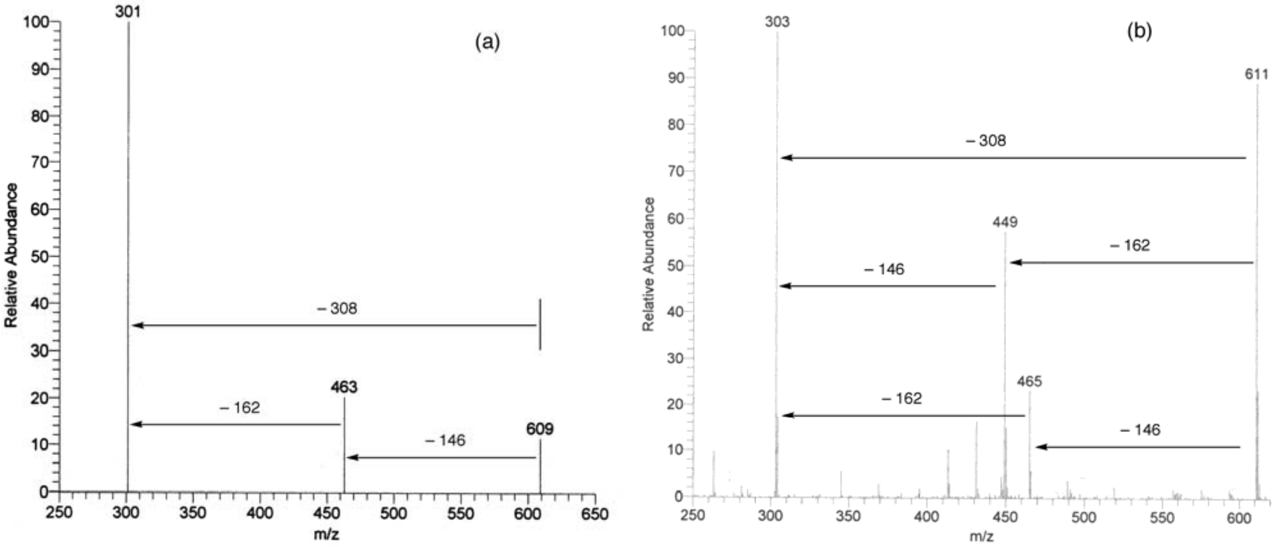


Figure 3 shows the UV spectra of quercetin (**10**), quercetin 3-*O*-glucoside, and quercetin 3,4'-di-*O*-glucoside recorded by a DAD unit [32]. Band I shifts about 20 nm to shorter wavelengths, with respect to quercetin, as a consequence of the hypsochromic effect caused by substitution at the 3 position. 3,4'-Substitution increases the hypsochromic shift, as shown in Figure 3. The UV spectra of flavanones generally show a strong absorption peak in the range 270-295 nm (Band II) and a shoulder of lower intensity, representing Band I (Figure 4) [31].

MS and MS-MS spectra of flavonoid glycosides have typical patterns, which depend mainly on the number or nature of the bound saccharides and their *C*- or *O*-glycosidic linkages [33]. Fragmentation of an *O*-glycoside starts from the cleavage of the *O*-sugar bond, and this behaviour is useful for identifying the aglycone [34]. The loss of fragments with well-defined mass from the pseudomolecular ion can provide precise information about the linked saccharide. Figure 5a presents the ESI-MS spectrum in negative mode of an *O*-disaccharide-substituted flavanone, *i.e.* hesperidin (hesperetin 7-*O*-rutinoside, **23**) [35]. The fragment *m/e* 463 was generated by the loss of one sugar unit (rhamnose) from the pseudomolecular ion $[M–H]^-$ (*m/e* 609). Subsequent loss of a second sugar unit (glucose) generates the ion *m/e* 301, which is easily assignable to the aglycone. The positive mode MS spectrum of the same compound **23** (Figure 5b) shows a different pattern of fragmentation. The pseudomolecular ion $[M+H]^+$ undergoes a partial rearrangement along a fragmentation pathway which is peculiar to this type of compounds. This leads to the loss of the "internal" sugar of the disaccharide (glucose, in the example described) demonstrated by the presence of the $[M+H–162]^+$ ion [36]. This fragmentation however is concomitant with the expected pathway, *i.e.* the loss of the rhamnose moiety.

Figure 5. Negative mode (a) and positive mode (b) ESI-MS spectra of a flavanone bearing a disaccharide as a substituent (hesperidin, **23**).



MS-MS experiments are very useful for identifying the aglycone of a given flavonoid. In Figure 6 appears the MS-MS spectrum in negative mode of the aglycone hesperetin (**1**) [35]. Fragmentation pattern analysis is highly diagnostic, and it is possible to obtain a precise structure elucidation of the aglycone by comparison with the literature data [37].

A *C*-glycoside flavonoid MS-MS spectrum focusing on the pseudomolecular ion $[M–H]^-$ presents again a typical fragmentation pattern, as shown in Figure 7 for diosmetin 6,8-di-*C*-glucoside (**32**). The fragmentation starts from the saccharide, and proceeds with the appearance of a well-defined series of characteristic ions: $[M–H–18]^-$, $[M–H–60]^-$, $[M–H–90]^-$, $[M–H–120]^-$, $[M–H–120–90]^-$, $[M–H–120–120]^-$ [38].

Figure 6. MS-MS spectrum in negative mode the aglycone hesperetin (**1**).

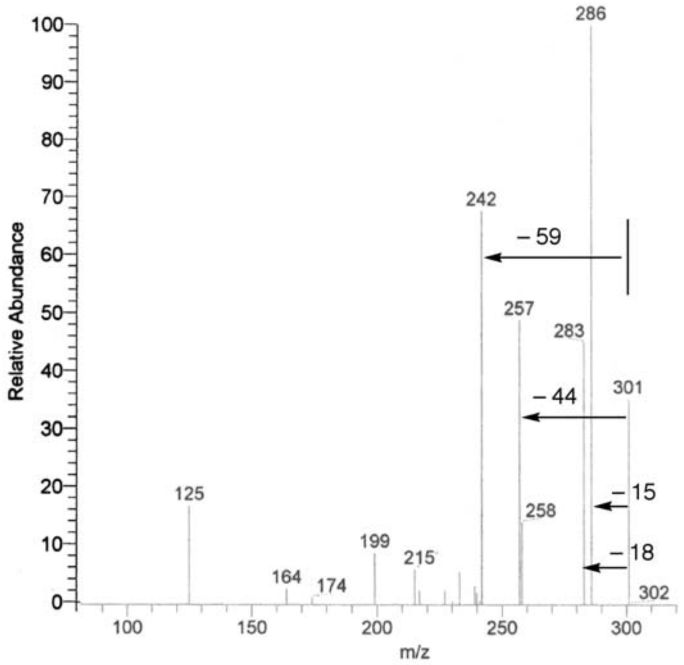
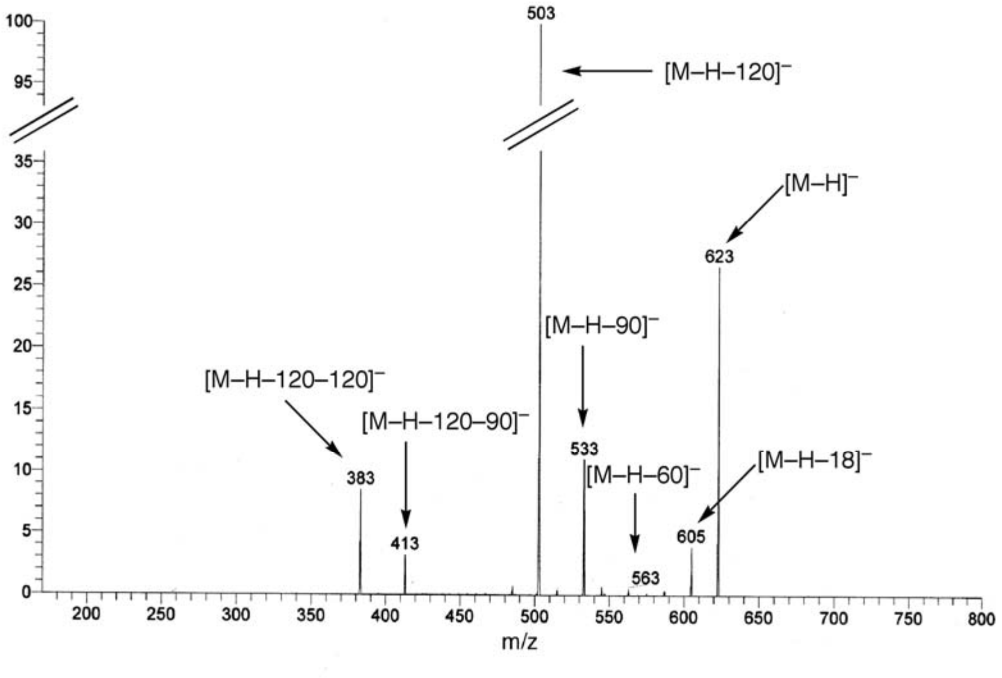
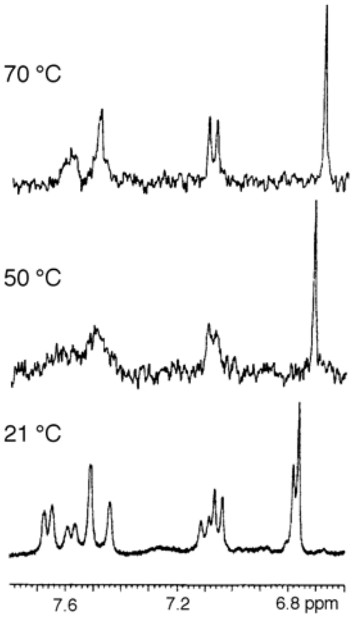


Figure 7. Negative mode MS-MS spectrum (focused on the pseudomolecular ion) of diosmetin 6,8-di-*C*-glucoside (**32**).



^{1}H-NMR spectra of *C*-glucoside flavonoids are particularly interesting because of the presence of rotamers resulting from hindered rotation around the *C*-glycosidic linkage. The two rotamers, often one major and the other minor, produce duplicated NMR peaks as a consequence of hindered rotation at room temperature on the NMR timescale [39]. Coalescence [40] of the signals may be easily achieved by variable temperature NMR experiments (VT-NMR), as shown in Figure 8, demonstrating that the two sets of signal belong to different conformers and not to different compounds. It is interesting to observe that the energy levels of the rotational isomers are often differently populated.

Figure 8. Variable temperature ^{1}H-NMR spectrum of 6,8-di-*C*-glucosyl diosmetin (**32**), recorded in DMSO-d_6 at 300 MHz. See ref. [33].



Sample preparation

Many analytical procedures require no preliminary separation of the flavonoid fraction, and analyses are performed directly on the crude juices [41]. Extraction with solvents of graded polarity [42] leads to the separation of flavonoids from the other components. Methanol [43], ethanol [44], acetone [45], water, ethyl acetate [46] and, to a lesser extent, propanol, dimethylformamide and combinations of these are frequently used for their extraction [47].

Hydrolysis of glycosides is a useful method to obtain structural elucidation and characterization information [48]. The rate of acid or basic hydrolysis of glycosides depends on acid or base strength, the nature of the sugar and the position of attachment to the flavonoid nucleus. In fact acid hydrolysis, which does not affect *C*-glycosides, is a good method to distinguish these derivatives from their *O*-glycosidic counterparts as the latter are quickly hydrolyzed [38b].

The extracts containing the flavonoids can be partially purified by using, for example, C18 Sep-Pak® cartridges [49]. Filtration [42c] has been used for flavonoid recovery from *Citrus*. Poor rates of recovery can be attributed to low solubility of certain flavonoids and also to absorption losses on the filtration medium. Addition of dimethylformamide to the juice has been used to improve the solubility of, for example, hesperidin, the major flavonoid component of sweet oranges and related species.

Although a significant number of publications on the analysis of flavonoid in *Citrus* juice have already appeared over the past two decades [50], there is still no standardized procedure available for sample preparation and extraction [42c].

Analytical approach to flavonoids determination

A number and variety of methods for the detection and quantification of flavonoid compounds in fruit have already been developed [51]. Several analytical procedures allow the simultaneous determination of the various kinds of flavonoid glycosides as flavanone-*O*-glycosides, flavone-*O*-glycosides, flavone-*C*-glucosides and polymethoxyflavones. Among the various techniques, we will focus on liquid chromatography (LC) and more recent coupled methods as LC-MS, and LC-MS-MS, with brief mention of less widely employed techniques (GC-MS, CE)

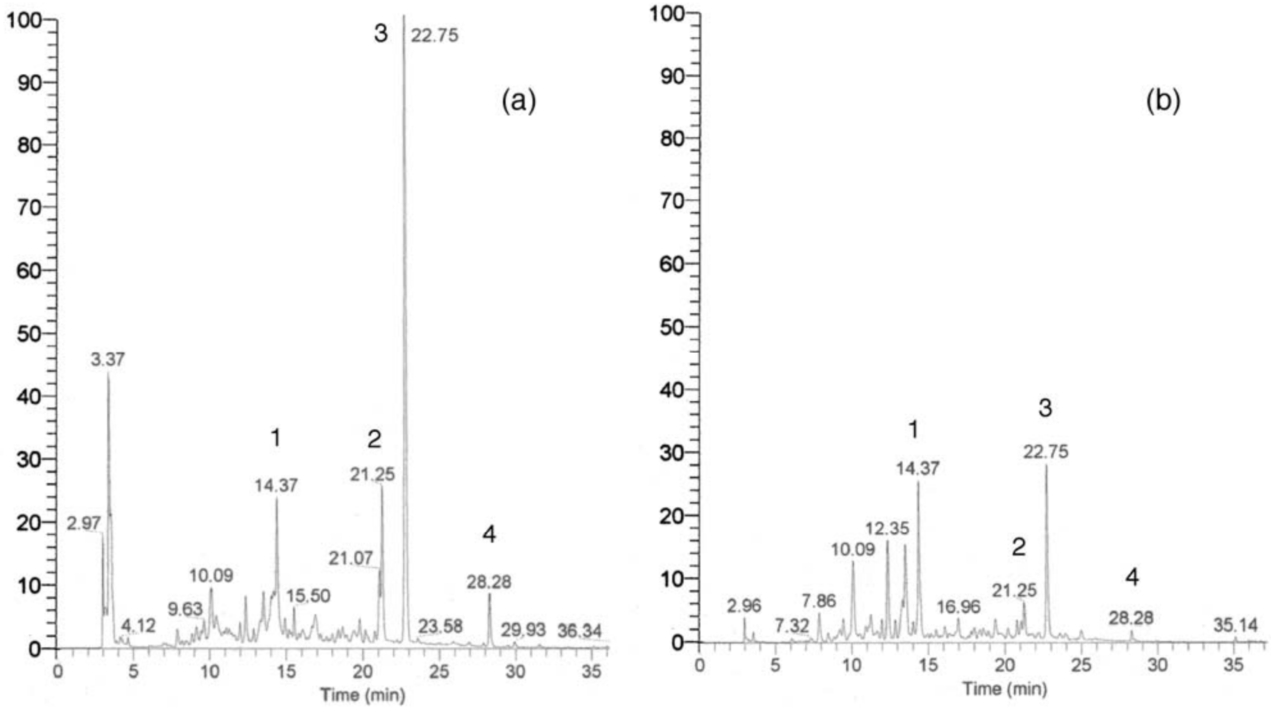
Gas chromatography (GC)

GC techniques are of limited application in flavonoids analysis as a consequence of the very low volatility of flavonoids, especially glycosylated ones. Usually flavonoids are transformed to more volatile derivatives by suitable derivatization [52].

High Performance Liquid Chromatography (HPLC)

HPLC methodologies represent, to date, the most widely used approach to the analysis of phenolics [53]. In most cases, HPLC techniques allow flavonoid profiles in juices to be obtained without the need for preliminary derivatization and sample preparation. Reversed-phase chromatography has been extensively employed for the separation of flavonoids on C8 or C18 columns [43, 52a, 53a,b, 54] with polar mobile phases, such as methanol, acetonitrile, tetrahydrofuran or acid solutions [55]. Gradient elution has often been used to obtain the profile of separated flavonoids [43, 53a,b, 56]. Under normal reversed-phase conditions, the more polar compounds are generally eluted first. Thus, diglycosides precede monoglycosides, which in turn precede aglycones. The classes of flavonoids that characterize *Citrus* species (flavanones, flavones, and, to a lesser extent flavonols/flavanols) have their maximum absorption at specific wavelength ranges: flavanones (280-290 nm), flavones (304-350 nm) and flavonols (352-385 nm). Simultaneously recorded DAD chromatograms of a red orange juice at two different wavelengths, for example, at 280 and 325 nm (Figure 9), highlights the difference in behaviour of flavanones compared to flavones under UV detection. In fact, flavanones narirutin, hesperidin, and didymin present intense peaks (marked as 2, 3, and 4, respectively, in Figure 9a) at 280 nm, whereas their relative absorptions decrease significantly at 325 nm (Figure 9b). Moreover, at 325 nm the flavone apigenin 6,8-di-*C*-glucoside shows an absorption peak (marked as 1), which is similar to the corresponding peak at 280 nm.

Figure 9. Simultaneously recorded DAD chromatograms of a red orange juice at two different wavelengths, for examples, at (a): 280 nm and (b): 325 nm.



Other chromatographic and miscellaneous methods.

Other techniques have been investigated for application to flavonoid analysis, although of less importance than the extensively applied HPLC. Thus, capillary electrochromatography method has been used to detect 7-*O*-glycosides in *Citrus* juices [57]. The same technique has been examined in the chiral separation of six diastereoisomeric flavanone-7-*O*-glycosides in lemon juice [58]. Flavonoid separation in orange juice has also been achieved by using Micellar Electrokinetic Chromatography [59].

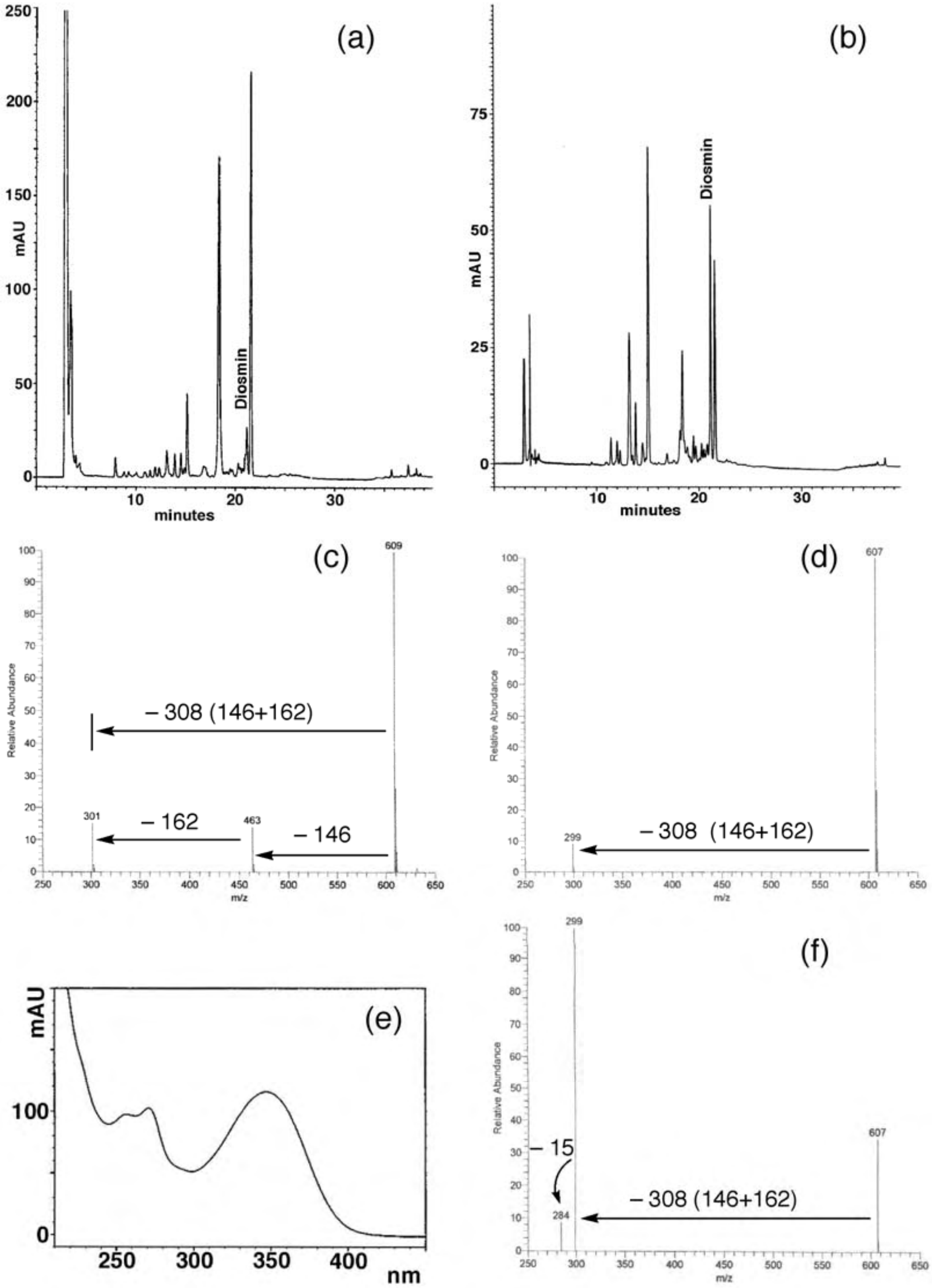
A different technique has been employed for the direct determination of naringin, making use of the formation of naringin-mercury complexes on the surface of a hanging mercury drop electrode. Cathodic stripping voltammetry makes it possible to determine of naringin between 0.1 and 40 mg/L [60].

Coupled methods

HPLC coupled with mass spectrometry (tandem HPLC-MS) [61] has often been used for the structural characterization of phenolics. The identification of phenolics collected after HPLC separation has also been carried out using fast atom bombardment mass spectrometry (FAB-MS) [62], electrospray ionization mass spectrometry (ESI-MS) [63] and atmospheric pressure chemical ionization (APCI-MS) [64]. In some cases, MS detection may provide enough data for a complete flavonoid structure analysis. However it is more generally used to determine molecular mass and to establish the nature of substituents between the A and the B rings. The ESI or APCI sources are very soft in the ionization and are commonly utilized to obtain TIC chromatograms and mass spectra in correspondence with each peak. Mass spectra, generated in positive mode, show pseudomolecular

$[M+H]^+$ ions together with other fragments depending on the voltage applied to the source. An acid (acetic or formic) is often added to mobile phases as a source of protons to assist ionization. Many flavonoids show low sensitivity in positive mode MS analysis and are therefore detected in negative mode. In this case, fragmentations start from pseudomolecular peak $[M–H]^–$.

Figure 10. HPLC-DAD-ESI-MS-MS analysis of a lemon juice sample. a) Chromatogram at 280 nm; b) chromatogram at 325 nm; c) positive mode MS spectrum of the peak at RT 21.1 (labeled as diosmin **43** in the chromatograms); d) negative mode MS spectrum of **43**; e) UV spectrum of **43**; f) negative mode MS-MS focused on the *m/e* 607 ion.



HPLC-diode array detection-electrospray mass spectrometry [65] has recently been used for a qualitative and quantitative determination of the flavonoid content of *Citrus* juices [30, 38a, 40, 66]. Often, one-course chromatographic analysis has been performed on crude juice. Two chromatograms at different wavelengths, and in correspondence with each peak UV spectrum along with full mass and MS-MS mass spectra can be recorded simultaneously. This approach has been successfully applied to problems involving trace analysis of *Citrus* flavanones, and can be expected to significantly improve the future development of LC-MS applications. Figure 10 provides an example of the HPLC-DAD-ESI-MS-MS analysis of a lemon juice, focusing on the peak corresponding to diosmin (**43**). Other interesting coupled methods are those based on HPLC separation coupled with electrochemical detection, being it either coulometric or amperometric [53m, 57a, 67].

Flavonoid composition of juices

The data reported are fairly heterogeneous as a result of the different techniques employed and the different units of measurement used by the various authors [68] (mg/100 mg juice, mg/100 mg lyophilized juice, mg/100 mL juice, mg/100 mg fresh product and so forth). Data were therefore corrected to obtain figures that could be compared and correlated. Data reported in μmol/L and mg/100 g were converted in mg/100 mL by taking into account the molecular weight of the compounds and/or the density of *Citrus* juices (from Brix data, assumed to be 1.045 mg/mL for freshly squeezed orange juice; 1.052 mg/mL for commercially processed orange juice, 1.044 mg/mL for fresh grapefruit, 1.057 mg/mL for commercial grapefruit, 1.040 mg/mL for lemon juice). A few Authors reported their results in mg/100 g of fresh weight [69]. These papers, although they will be cited in the relevant sections, have been omitted from statistical calculations.

Also, many papers focus on only a few of the flavonoids present, usually the most abundant ones, whereas several flavonoids have only recently been identified, owing to the ever-increasing quality of instruments and analytical methodologies. The Tables of composition are structured as follows: the abbreviated name of the compound found, the mean value, along with statistical data (standard deviation, median, minimum-maximum range) and the number of values found in literature (marked as N). Another preliminary regards the classification of different plants. These will be discussed in line with Tanaka’s classification of *Citrus* species [70].

Orange

The genus *Citrus* comprises several orange species – *Citrus sinensis* (sweet orange), *Citrus aurantium* (sour oranges), *Citrus reticulata* (tangerine or mandarin) – and their hybrids – tangors, which are orange-tangerine hybrids, and tangelos, which are tangerine-grapefruit or tangerine-pummelo hybrids. Many of these species or hybrids can have different varieties. Sweet oranges, for instance, have several subclasses: Common, Navel, Valencia, and Blood, to name a few of the most widespread. Tangerine or mandarin species are also *C. clementina*, *C. deliciosa*, *C. nobilis*, and *C. unshiu*. In addition, several hybrids have other hybrids as parents, making the classification more difficult.

Table 6 shows the data collected for the flavonoid content of hand-squeezed juices for *C. sinensis*. The most abundant component in sweet orange juices, regardless of variety, is by far hesperidin (**23**, 28.6 mg/100 mL), followed by narirutin (**25**, 5.2 mg/100 mL) [71]. Also, didymin (**21**, 1.89 mg/100 mL) has been detected as being a significant component of *C. sinensis* juice [71a]. These are all flavanone-*O*-glycosides, which account for most of the flavonoid content in juice. However, more recently 6,8-di-*C*-glucosyl-apigenin (vicenin-2, **30**, 5.72 mg/100 mL) has been shown to be present in considerable amounts [38a, 71e].

Table 6. Flavonoid composition of *C. sinensis* juice (expressed in mg/100 mL).

		Mean	SD	Median	MIN	MAX	N	Ref.
Flavanones								
Didymin	**21**	1.89	0.92	1.60	0.80	3.10	7[a]	[71a]
Eriocitrin	**22**	0.31	0.18	0.29	0.11	0.67	7[a]	[71a]
Hesperidin	**23**	28.6	11.9	28.0	3.51	55.2	44[b]	[71]
Narirutin	**25**	5.2	3.1	4.2	0.55	14.2	44[b]	[71]
Flavones								
Neoeriocitrin	**27**	0.59	–	–	–	–	1[c]	[71d]
Poncirin	**28**	1.04	0.78	1.04	0.49	1.59	2[d]	[71d]
6,8-di-*C*-Glu-Apigenin	**30**	5.72	2.02	5.00	4.15	8	3[e]	[38a, 71e]
6,8-di-*C*-Glu-Diosmetin	**32**	0.35	0.14	0.35	0.25	0.45	2[f]	[38a]
Rhoifolin	**40**	0.05	–	–	–	–	1[c]	[71d]
Isorhoifolin	**41**	0.07	–	–	–	–	1[c]	[71d]
Diosmin	**43**	0.09	–	–	–	–	1[g]	[71d]
Neodiosmin	**44**	0.08	–	–	–	–	1[g]	[71d]
Polymethoxyflavones								
Heptamethoxyflavone	**15**	0.08	0.06	0.08	0.04	0.12	2[d]	[71d,f]
Nobiletin	**17**	0.33	0.19	0.33	0.19	0.46	2[d]	[71d,f]
Sinensetin	**18**	0.37	–	–	–	–	1[g]	[71d,f]
Tangeretin	**19**	0.04	0.04	0.04	0.01	0.07	2[d]	[71d,f]
Aglycones								
Taxifolin	**3**	0.03	–	–	–	–	1[c]	[71d]
Acacetin	**6**	0.03	–	–	–	–	1[c]	[71d]

[a] Var. Salustiana, Maltaise, Sanguinelli, Valencia, Navel, Washington Sanguinelli, Thomson. [b] Var. Salustiana, Hamlin, Maltaise, Shamouti, Sanguinelli, Valencia, Pera, Cara-cara, Navel, Washington Sanguinelli, Thomson. Natal, Baìa, Lima. [c] Var. Navel. [d] Var. Valencia, Navel. [e] Var. Navel, Moro, Tarocco. [f] Var. Tarocco. [g] Var. Valencia.

A further fourteen components have been found by different Authors, mostly in low amounts (<0.6 mg/100 mL) [72]. Among these, it is worth mentioning diosmin (**43**, 0.09 mg/100 mL) and neodiosmin (**44**, 0.08 mg/100 mL), reported by Kawaii *et al.* [71d], which, along with the flavone 6,8-di-*C*-glucosyl-diosmetin (**32**, 0.35 mg/100 mL) [38a], are generally associated with lemon juices (*vide infra*). Four different polymethoxyflavones have also been described, namely sinensetin (**18**, 0.37 mg/100 mL) [71c,d] nobiletin (**17**, 0.33 mg/100 mL) [71c,d], tangeretin (**19**, 0.04 mg/100 mL) [71c,d] and 3,3',4',5,6,7,8-heptamethoxyflavone (**15**, 0.08 mg/100 mL) [71c,d]. Polymethoxyflavones have been reported over the years as minor components of orange juice [71d,f]. However, a recent paper, in which the juice samples were prepared by isolating of the endocarp, demonstrated that they are virtually absent in juice. Therefore, it is probable that in previous reports they were found as a result of sample preparation procedures and derived from the essential oils fraction of the peel [30, 73].

In general, the data where the specific *C. sinensis* variety analyzed is reported, show that different varieties present approximately the same flavonoid composition pattern. There is little or no significant quantitative difference between the various juices, the var. Shamouti, Sanguinelli and Pera being the richest, and Maltaise and Valencia the poorest.

Table 7 shows the results obtained from unspecified commercial orange juice samples (*i.e.* commercial products where the exact species and/or variety of the oranges in not given, or samples made from frozen concentrates) [30, 47b, 49, 57b, 71e, 74].

Commercial orange juices present a similar composition to freshly squeezed ones, with the appearance of some unexpected compounds. Naringin (**24**, 2.13 mg/100 mL) and diosmin (**43**, 3.46 mg/100 mL) hint at the possibility that some of the samples analyzed are not pure orange juices [75]. Diosmin is typically found in lemon juice, whereas naringin and naringenin (**2**, 0.8 mg/100 mL) are components typical of grapefruit juice (*vide infra*).

As in the case of hand-squeezed juices, the presence of PMFs in variable quantities suggests that they could be essentially derived from the flavedo and confirm that the amounts of PMFs found in industrial juices are a consequence of the pressing process used [30].

Table 7. Flavonoid composition of commercial sweet orange juice (mg/100 mL).

		Mean	SD	Median	MIN	MAX	N	Ref.
Flavanones								
Didymin	**21**	1.89	0.56	1.78	1.14	3.53	32	[30, 74b,d]
Hesperidin	**23**	37.5	19.2	39.1	4.45	76.3	63	[30, 47b, 49, 57b, 74b-f]
Naringin	**24**	2.13	3.01	0.34	0.20	7.54	9	[49, 74a,c,e]
Narirutin	**25**	5.9	1.6	6.3	2.95	9.78	46	[30, 47b, 49, 57b, 74b-d]
Neohesperidin	**26**	0.95	0.32	1.10	0.53	1.29	5	[74e]
Flavones								
6,8-di-*C*-Glu-Apigenin	**30**	4.16	1.37	3.95	2.78	8.00	13	[71e]
Diosmin	**43**	3.46	1.92	2.60	0.79	7.20	11	[74c]
Polymethoxyflavones								
Quercetogetin	**14**	0.04	0.01	0.04	0.03	0.05	4	[30, 74d]
Heptamethoxyflavone	**15**	0.05	0.03	0.05	0.03	0.07	2	[30]
Nobiletin	**17**	0.26	0.07	0.26	0.18	0.34	4	[30, 74d]
Sinensetin	**18**	0.24	0.09	0.22	0.17	0.36	4	[30, 74d]
Tangeretin	**19**	0.04	0.02	0.05	0.02	0.06	4	[30, 74d]
Aglycones								
Naringenin	**2**	0.8	–	–	–	–	1	[74f]
Isoscutellarein	**7**	0.05	0.04	0.05	0.02	0.08	2	[30]

Table 8 contains literature data on the flavonoid content of *C. reticulata* juice [71a,d, 74d]. Mandarin juice is quite similar to sweet orange juice, since they are characterized by the same distinctive flavanones. Hesperidin (**23**, 24.3 mg/100 mL) is again the main component, followed by narirutin (**25**, 3.92 mg/100 mL) and didymin (**21**, 1.44 mg/100 mL), with neohesperidin and naringin being virtually absent [76], confirming that *C. sinensis* and *C. reticulata* are closely related. However, the ratios between the flavanone and polymethoxyflavones fractions in sweet orange and mandarin are slightly different. On this basis, Ooghe *et al.* [77] established a procedure to detect the addition of mandarin to orange juices in commercial products by HPLC equipped with a photodiode array (PDA).

Table 8. Flavonoid composition of *C. reticulata* juice (mg/100 mL).

		Mean	SD	Median	MIN	MAX	N	Ref.
Flavanones								
Didymin	**21**	1.44	1.31	1.60	0.05	3.10	7[a]	[71a,d, 74d]
Eriocitrin	**22**	0.31	0.18	0.31	0.09	0.59	5[a]	[71a,d, 74d]
Hesperidin	**23**	24.3	18.2	28	0.81	45.8	7[a]	[71a,d, 74d]
Narirutin	**25**	3.92	3.10	4.80	0.12	9.00	7[a]	[71a,d, 74d]
Neoeriocitrin	**27**	0.05	0.01	0.05	0.04	0.06	2[b]	[71d]
Polymethoxyflavones								
Quercetogetin	**14**	0,06	–	–	–	–	1[c]	[74d]
Heptamethoxyflavone	**15**	0.07	–	–	–	–	1[d]	[74d]
Nobiletin	**17**	0.23	0.10	0.20	0.15	0.35	3[e]	[71d, 74d]
Sinensetin	**18**	1.05	–	–	–	–	1[c]	[74d]
Tangeretin	**19**	0.26	0.23	0.19	0.07	0.52	3[e]	[71d, 74d]
Aglycones								
Acacetin	**6**	0.02	–	–	–	–	1[f]	[71d]

[a] Var. Ortanique, Clementine, Mineola, Fortuna, Dancy. [b] Var. Clementine, Dancy. [c] Var. Ortanique. [d] Var. Clementine. [e] Var. Ortanique, Clementine, Dancy. [f] Var. Dancy.

Table 9 contains data reported on *C. clementina* juice composition [38a, 71b, 74c]. *C. clementina* is taxonomically related to both *C. reticulata*, and *C. sinensis*, and like the latter species presents a high concentration of hesperidin (**23**, 39.9 mg/100 mL) as its main component, followed by narirutin (**25**, 4.64 mg/100 mL). Nogata *et al.* [69a] reported the presence of rutin (**39**) in *C. clementina* juice. Both 6,8-di-*C*-glucosyl apigenin (**30**, 0.5 mg/100 mL) and 6,8-di-*C*-glucosyl diosmetin (**32**, 0.2 mg/100 mL) are present, but in negligible amounts.

Table 9. Flavonoid composition of *C. clementina* juice (mg/100 mL).

		Mean	SD	Median	MIN	MAX	N	Ref.
Flavanones								
Hesperidin	**23**	39.9	29.4	34.9	5.21	86.1	8	[71b,74c]
Naringin	**24**	0.08	0.03	0.08	0.05	0.12	4	[71b]
Narirutin	**25**	4.64	–	–	–	–	1	[71b]
Flavones								
6,8-di-*C*-Glu-Apigenin	**30**	0.5	–	–	–	–	1	[38a]
6,8-di-*C*-Glu-Diosmetin	**32**	0.2	–	–	–	–	1	[38a]
Diosmin	**43**	1.25	0.51	1.26	0.67	2.12	7	[74c]

As far as *C. deliciosa* is concerned, only two papers which would fit the criteria for discussion in this review were found (Table 10): Dhuique-Mayer *et al.* [71b] report quantification of hesperidin (**23**, 76.7 mg/100 mL) and narirutin (**25**, 3.72 mg/100 mL), whereas Caristi *et al.* [38a] report on the 6,8-di-*C*-glucosyl apigenin (**30**, 2.5 mg/100 mL) and 6,8-di-*C*-glucosyl diosmetin (**32**, 0.7 mg/100 mL) content.

Table 10. Flavonoid composition of *C. deliciosa* juice (mg/100 mL).

		Values	N	Ref.
Flavanones				
Hesperidin	**23**	0.15	1	[71b]
Narirutin	**25**	1.97	1	[71b]
Flavones				
6,8-di-*C*-Glu-Apigenin	**30**	2.5	1	[38a]
6,8-di-*C*-Glu-Diosmetin	**32**	0.7	1	[38a]

Contrary to sweet orange, mandarin and their closely related species, *C. aurantium* are not generally suitable for direct consumption. Indeed, sour oranges are mostly used for their essential oils, which are employed by the perfume and food industries as flavoring agents. They are also used in herbal medicine and in dietary supplements as an aid to fat loss and as an appetite suppressant. Kawaii *et al.* [71d] report on the composition of *C. aurantium* (Table 11). The juice composition is very different from sweet orange, but it is quite similar to that of grapefruit, being rich in naringin (**24**, 1.96 mg/100 mL), neohesperidin (**26**, 0.87 mg/100 mL) and neoeriocitrin (**27**, 0.77 mg/100 mL), with hesperidin (**23**) being virtually absent [53j, 69b]. Ooghe *et al.* [75] optimized a method to detect the addition of as little as 2% of sour orange juice to commercial sweet orange juice, by measuring the amount of naringin and neohesperidin.

Table 11. Flavonoid composition of *C. aurantium* juice (mg/100 mL).

		Values	N	Ref.
Flavanones				
Naringin	**24**	1.97	1	[71d]
Neohesperidin	**26**	0.87	1	[71d]
Neoeriocitrin	**27**	0.77	1	[71d]
Poncirin	**28**	0.73	1	[71d]
Flavones				
Diosmin	**43**	0.15	1	[71d]
Polymethoxyflavones				
Nobiletin	**17**	0.2	1	[71d]
Tangeretin	**19**	0.08	1	[71d]
Aglycones				
Kaempferol	**9**	0.14	1	[71d]

Lemon

Lemon (*C. limon*) juice [38a, 40, 71d, 78, 79] is characterized by the presence of significant amounts of the flavanones, hesperidin (**23**, 20.5 mg/100 mL) and eriocitrin (**22**, 16.7 mg/100 mL), as depicted in Table 12. Lemon juice is also quite rich in flavones: diosmin has been recognized as one of the main flavonoid components of this juice. Recently, however, more data published on di-*C*-glucosyl flavones showed them to be present in significant amounts. *C. limon* juice has been reported to be rich in diosmetin 6,8-di-*C*-glucoside (**32**, 4.95 mg/100 mL), and also contains apigenin di-*C*-glucoside (**30**, 1.17 mg/100 mL). It is interesting to observe that the ratio between the two flavones is inverted with respect to sweet orange juice (see Table 6) [38a]. Commercial lemon juices [57b, 74c,e], as described in Table 13, reflect the composition of the hand-squeezed ones, with the notable exception of hesperidin (**23**, 7.07 mg/100 mL).

Table 12. Flavonoid composition of *C. limon* juice (mg/100 mL).

		Mean	SD	Median	MIN	MAX	N	Ref.
Flavanones								
Eriocitrin	**22**	16.7	10.3	16.55	1.67	39.1	10[a]	[40, 71d, 78]
Hesperidin	**23**	20.5	12.4	18.85	3.84	41	10[a]	[40, 71d, 78]
Flavones								
6,8-di-*C*-Glu-Apigenin	**30**	1.17	0.25	1.05	1	1.45	3[b]	[38a]
6,8-di-*C*-Glu-Diosmetin	**32**	4.95	0.88	5	4.05	5.8	3[b]	[38a]
7-*O*-Rut-Luteolin	**36**	3.93	2.14	3.5	1.5	6.5	6[c]	[78]
Diosmin	**43**	3.12	1.66	3.65	0.51	5.1	10[a]	[40, 71d, 80]
Aglycones								
Luteolin	**8**	0.08	–	–	–	–	1[d]	[71d]

[a] Var. Fino, Verna, Eureka, Femminello Comune, Monachello, Interdonato. [b] Var. Femminello Comune, Monachello, Interdonato. [c] Var. Fino, Verna, Eureka. [d] Var. Eureka.

Table 13. Flavonoid composition of commercial lemon juice (mg/100 mL).

		Mean	SD	Median	MIN	MAX	N	Ref.
Flavanones								
Eriocitrin	**22**	16.0	–	–	–	–	1	[57b]
Hesperidin	**23**	7.07	9.02	3.06	1.59	23.08	5	[57b, 74c]
Naringin	**24**	0.38	–	–	–	–	1	[74c]
Neohesperidin	**26**	1.45	–	–	–	–	1	[74e]
Flavones								
Diosmin	**43**	1.04	0.67	1.12	0.16	1.78	4	[74c]

There are several *Citrus* species commonly referred to as “limes”, depending mostly on geographical distinction: *C. latifolia*, *C. limetta*, *C. limettoides*, *C. hystrix*, *C. ichangensis*. The most representative species, and the one that is more correctly defined as “lime” is however *C. aurantifolia*. Table 14 describes the limited data collected for lime juice [71d]. Its composition closely resembles the flavonoid content of lemon juice, at least as far as the distribution is concerned. Hesperidin (**23**, 1.77 mg/100 mL) is again the main flavanone component, followed by eriocitrin (**22**, 0.29 mg/100 mL). On the other hand, total flavonoid content has been found to be rather lower than for *C. limon*.

Bergamot

C. bergamia is a species of uncertain origins, believed to derive from a cross between lemon and sweet orange (*C. limon X C. sinensis*), or sour orange and citron or grapefruit (*C. aurantium X C. medica*, the latter not discussed in this review, or *C. aurantium X C. paradisi*). It has been produced for centuries now, for the most part only on a very small coastal strip in the province of Reggio Calabria, Italy. Although it was prized almost exclusively for its essential oils, and their application in cosmetic industry (perfumes), its juice recently has been found to be quite rich in flavonoids components [38a, 71d].

Table 14. Flavonoid composition of *C. aurantifolia* juice (mg/100 mL).

		Mean	SD	Median	MIN	MAX	N	Ref.
Flavanones								
Eriocitrin	**22**	0.29	–	–	–	–	1[a]	[71d]
Hesperidin	**23**	1.77	0.35	1.77	1.52	2.02	2[b]	[71d]
Neoeriocitrin	**27**	0.01	–	–	–	–	1[c]	[71d]
Flavones								
Diosmin	**43**	0.08	–	–	–	–	1[c]	[71d]
Polymethoxyflavones								
Heptamethoxyflavone	**15**	0.12	–	–	–	–	1[c]	[71d]
Natsudaidain	**16**	0.04	–	–	–	–	1[c]	[71d]
Nobiletin	**17**	0.52	–	–	–	–	1[c]	[71d]
Tangeretin	**19**	0.18	–	–	–	–	1[c]	[71d]
Aglycones								
Taxifolin	**3**	0.04	–	–	–	–	1[c]	[71d]
Luteolin	**8**	0.61	–	–	–	–	1[a]	[71d]

[a] Var. Tahiti. [b] Var. Tahiti, Sweet Lime. [c] Var. Sweet Lime.

Hand-squeezed juice is characterized by the presence of good amounts of poncirin (**28**, 6.41 mg/100 mL) and naringin (**24**, 2.23 mg/100 mL), closely followed by neohesperidin (**26**, 1.60 mg/100 mL) and neoeriocitrin (**27**, 1.38 mg/100 mL). Diosmetin 6,8-di-*C*-glucoside (**32**, 3.95 mg/100 mL) and apigenin 6,8-di-*C*-glucoside (**30**, 4.53 mg/100 mL) are present in almost equal amounts, hinting that *C. bergamia* may indeed descend from a hybrid of *C. limon* and *C. sinensis.*

Table 15. Flavonoid composition of *C. bergamia* juice (mg/100 mL).

		Mean	SD	Median	MIN	MAX	N	Ref.
Flavanones								
Naringin	**24**	2.23	–	–	–	–	1	[71d]
Neohesperidin	**26**	1.60	–	–	–	–	1	[71d]
Neoeriocitrin	**27**	1.38	–	–	–	–	1	[71d]
Poncirin	**28**	6.41	–	–	–	–	1	[71d]
Flavones								
6,8-di-*C*-Glu-Apigenin	**30**	4.53	0.58	4.45	4.0	5.15	3[a]	[38a]
6,8-di-*C*-Glu-Diosmetin	**32**	3.95	0.71	4.05	3.20	4.60	3[a]	[38a]
Rhoifolin	**40**	0.37	–	–	–	–	1	[71d]
Diosmin	**43**	0.39	–	–	–	–	1	[71d]
Neodiosmin	**44**	0.3	–	–	–	–	1	[71d]

[a] Var. Fantastico, Femminello, Castagnaro.

Industrial bergamot juice has been produced for years but as a mere byproduct by the essential oils industry. It has been found to be extremely rich in flavonoids [66]. Neoeriocitrin (**27**, 27.6 mg/100 mL), naringin (**24**, 26.1 mg/100 mL) and neohesperidin (**26**, 22.1 mg/100 mL) are the most abundant flavanones, whereas apigenin di-*C*-glucoside (**30**, 6.22 mg/100 mL), diosmetin di-*C*-glucoside (**32**, 4.38 mg/100 mL), rhoifolin (**40**, 6.06 mg/100 mL) and chrysoeriol 7-*O*-neohesperidoside (**42**, 4.89

mg/100 mL) are the most plentiful glycosyl flavones. Bergamot juice has yet to find an application in the food industry, despite its extremely high flavonoid content.

Table 16. Flavonoid composition of commercial bergamot juice (mg/100 mL).

		Mean	N	Ref.
Flavanones				
Eriocitrin	**22**	1.45	1[a]	[66]
Naringin	**24**	26.1	1[a]	[66]
Neohesperidin	**26**	22.1	1[a]	[66]
Neoeriocitrin	**27**	27.6	1[a]	[66]
Flavones				
6,8-di-*C*-Glu-Luteolin	**29**	0.69	1[a]	[66]
6,8-di-*C*-Glu-Apigenin	**30**	6.22	1[a]	[66]
6,8-di-*C*-Glu-Chrysoeriol	**31**	0.66	1[a]	[66]
6,8-di-*C*-Glu-Diosmetin	**32**	4.38	1[a]	[66]
4'-*O*-Glu Rhoifolin-	**33**	0.81	1[a]	[66]
7-*O*-Neohesp-4'-Glu-Chrysoeriol	**34**	1.24	1[a]	[66]
6-*C*-Glu-Apigenin	**35**	0.49	1[a]	[66]
8-*C*-Glu-Chrysoeriol	**37**	0.75	1[a]	[66]
8-*C*-Glu-Diosmetin	**38**	0.82	1[a]	[66]
Rhoifolin	**40**	6.06	1[a]	[66]
7-*O*-Neohesp-Chrysoeriol	**42**	4.89	1[a]	[66]
Neodiosmin	**44**	2.3	1[a]	[66]

[a] Juices from mixed pressing of Fantastico, Femminello and Castagnaro varieties.

Grapefruit

C. paradisi juice [57a, 71d, 81] can generally be found in three different varieties, red, pink and white, their color depending on the presence (or absence) of lycopene [82]. It has received a lot of attention over the past few years, since it has been found to possess the ability to increase the bioavailability of a number of drugs [81c, 83]. Its main component, as described in Table 17, is the flavanone naringin (**24**, 23.0 mg/100 mL) and also naringenin (**2**, 2.70 mg/100 mL), its aglycone, which has always been recognized to be a distinctive component of grapefruit juices. Narirutin is also present in good amounts (**25**, 7.60 mg/100 mL). Generally speaking, white grapefruit juice is slightly richer in flavonoids than the pink and red varieties.

Commercial grapefruit juices [47b, 57b, 60, 74b-e, 81a,c, 84] tend to be even more concentrated than their hand-squeezed equivalents. This results in an even higher concentration of the main components, naringin (**24**, 43.5 mg/100 mL), naringenin (**2**, 4.20 mg/100 mL) and narirutin (**25**, 9.90 mg/100 mL). Naringin is one of the species responsible for the bitter taste of grapefruit juice [28]. Several Authors have worked on optimizing debittering procedures based on the removal of naringin [7a, 81b, 85].

Table 17. Flavonoid composition of *C. paradisi* juice (mg/100 mL).

		Mean	SD	Median	MIN	MAX	N	Ref.
Flavanones								
Didymin	**21**	0.30	0.04	0.30	0.27	0.33	2[a]	[81d]
Eriocitrin	**22**	0.41	0.19	0.41	0.27	0.54	2[a]	[81d]
Hesperidin	**23**	0.93	0.58	0.87	0.25	1.79	6[a]	[57a, 71d, 81a,b,d]
Naringin	**24**	23.0	12.8	21.9	4.5	60.2	19[a]	[57a, 71d, 81]
Narirutin	**25**	7.60	5.80	7.70	2.50	17.0	5[a]	[71d, 81b,d]
Neohesperidin	**26**	1.21	0.35	1.28	0.67	1.58	5[a]	[71d, 81b,d]
Neoeriocitrin	**27**	0.32	0.02	0.32	0.30	0.33	2[b]	[71d]
Poncirin	**28**	1.26	0.35	1.30	0.85	1.58	4[a]	[71d, 81d]
Flavones								
Rutin	**39**	3.26	–	–	–	–	1	[57a]
Rhoifolin	**40**	0.28	–	–	–	–	1[c]	[71d]
Polymethoxyflavones								
Heptamethoxyflavone	**15**	0.06	0.07	0.06	0.01	0.11	2[b]	[71d]
Nobiletin	**17**	0.15	0.04	0.15	0.12	0.17	2[b]	[71d]
Tangeretin	**19**	0.12	–	–	–	–	1[c]	[71d]
Aglycones								
Hesperetin	**1**	0.74	–	–	–	–	1	[57a]
Naringenin	**2**	2.70	2.68	1.70	0.98	8.00	6	[57a, 81c]
Taxifolin	**3**	0.16	–	–	–	–	1[d]	[71d]
Quercetin	**10**	0.19	0.03	0.19	0.17	0.21	2[b]	[71d]

[a] Var. Red, Pink, White. [b] Var. Pink, White. [c] Var. White. [d] Var. Pink.

Table 18. Flavonoid composition of commercial grapefruit juice (mg/100 mL).

		Mean	SD	Median	MIN	MAX	N	Ref.
Flavanones								
Didymin	**21**	0.8	0.5	0.8	0.0	1.7	11[a]	[74d, 84]
Hesperidin	**23**	2.8	3.9	1.6	0.2	16.4	15[a]	[74c-e, 84]
Naringin	**24**	43.5	23.3	38.8	4.8	119.7	102[b]	[47b, 57b, 60, 74b-e, 81a,c, 84]
Narirutin	**25**	9.90	4.2	10.2	2.3	18.8	17[a]	[47b, 57b, 74b,d, 84]
Neohesperidin	**26**	0.8	0.5	0.9	0.67	2.0	18[a]	[74b,d,e, 84]
Poncirin	**28**	1.2	0.7	1.2	0.1	2.4	16[a]	[74b,d, 84]
Flavones								
Diosmin	**43**	0.80	0.8	0.8	0.20	1.40	2.0	[74c]
Aglycones								
Naringenin	**2**	4.20	4.00	3.30	0.40	16.2	44[a]	[81c]
Quercetin	**10**	0.6	0.2	0.6	0.2	0.9	7[a]	[74b, 84]

[a] Var. Red, Pink. [b] Var. Red, Pink, White.

Conclusions

The beneficial effects of flavonoids on human health is universally accepted nowadays. *Citrus* fruits and juices remain one of the most readily-available dietary sources for their intake. An in-depth knowledge of their composition and their action has still to be achieved. However, over the period of time examined in this review, researchers are progressing at an ever-increasing pace.

In the first part, a survey of the methodologies employed in *Citrus* juices analysis was presented. Analytical methods are improving in their ability to detect and quantify simultaneously the largest possible number of compounds, especially the health-beneficial phenolics.

In the second part, a collection of data from recent literature was presented and critically analyzed. It should be noted that for most *Citrus* species, the amount of data available is still scarce, Thus there is still an intense effort that must be invested in order to obtain further information on the phenolic content of many of the less studied species.

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